

## SCIENCE FOR CERAMIC PRODUCTION

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### CREEP RUPTURE STRENGTH OF MAGNESIUM OXIDE BASED DENSE POLYCRYSTALLINE CERAMIC AT TEMPERATURES TO 1600°C

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Experimental data on the determination of the creep rupture strength (time and strain to rupture) of densely sintered periclase ceramic at temperatures to 1600°C under loads 20 – 50 MPa are presented. It is shown that the durability of materials can be determined from measurements of the rate of steady creep below the rupture level.

**Key words:** densely sintered ceramic, periclase, durability, creep, time to rupture, rupture strain.

The creep rupture strength (sometimes called durability) is usually understood to be the time to rupture  $\tau_r$  of a material under a constant mechanical load in creep tests [1]. Thus, it gives an idea about the service life of ceramic at high temperatures. Another important characteristic of the process is the strain  $\dot{\epsilon}_r$  of a sample: some authors view strain as a signal indicating impending rupture. Here, it is assumed that the processes responsible for strain and rupture during creep are interrelated. Otherwise it would be impossible to explain the formation of embryonic cracks and their propagation under stresses much weaker than the theoretical strength of materials. Thus, the time to rupture  $\tau_r$  of metals and alloys is inversely proportional to their rate of steady creep  $\dot{\epsilon}$ , i.e.,  $\tau_r = \text{const}$  [1].

A large number of empirical and theoretical equations have been proposed for the creep rupture strength that relate the main criterion  $\tau_r$  (time to rupture) with the applied stress  $\sigma$  and temperature  $T$  [1]. They describe the results of measurements only in limited ranges of variation of the variables. Functions with inflections are usually obtained in wide intervals: power-law functions of the stress and exponential functions of the temperature  $\tau_r = A\sigma^n$  and  $\tau_r = B \exp(bT)$ , where  $A$ ,  $n$ ,  $B$  and  $b$  are empirical coefficients. Aside from these relations, so-called parametric relations have also been proposed:  $\tau_r = f(\sigma, T)$ . For  $\sigma = \text{const}$  they become functions of

the temperature and for  $T = \text{const}$  functions of the force, but their applicability is also limited.

The results of a study of strain and rupture of polycrystalline oxide ceramic at temperatures to 1600°C are discussed in [2, 3]. It is shown that above the brittle – plastic transition temperature the main mechanism of strain in such materials is diffusion-viscous flow with all its varieties (dislocation climb, boundary motion), which are also due to diffusion of atoms (ions) along vacant nodes or interstices. Previously, this mechanism was also established for higher temperatures [4, 5], where only steady creep is observed. At the same time nonstationary creep associated with the presence of stresses and nonequilibrium defects formed at high annealing temperatures and ‘quenched’ on cooling was also found at the lower bound of the temperature interval studied in [3]. In high-temperature tests the deformation associated with the relaxation of these imperfections of the crystal lattice is superposed on the creep strain and thereby results in the appearance of a nonstationary period [6]. The present work is a continuation of [2, 3] concerning the determination of the creep rupture strength of polycrystalline ceramic and analysis of the results obtained.

The aim of the present article is to expound the results of a determination of the indicators of creep rupture strength of dense magnesium-based ceramic at temperature to 1600°C and to establish a relation between these indicators and the characteristics of creep.

The experiments on measuring strain versus time were performed with constant stresses in the range 20 – 50 MPa. The testing procedure is described in [2, 3] and the characteristics of the samples are given in Table 1. We note that for the

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samples P-12 the experimental temperature range lies above the brittle – ductile transition, since their strength decreases monotonically with heating. At the same time, for the samples P-25 the brittle – ductile transition temperature falls inside the experimental range, since the strength reaches its maximum value at 1450°C. Ordinarily, this occurs in the presence of plasticity [7].

The experimental samples showed very small strain. For this reason, to obtain reliable data the applied loads were close to the ultimate strength of the ceramic. The effect of the testing conditions and crystal size on the durability indicators (time and strain to rupture) were determined from the data obtained.

The dependence of  $\tau_r$  on the stress  $\sigma$  for periclase ceramic is presented in Fig. 1. In the experimental temperature range the time to rupture decreases with increasing stress. The slope of the straight lines in logarithmic coordinates remains virtually constant; therefore, the mechanism of the process does not change with temperature. The dependence has the form  $\tau_r = A\sigma^n$  with  $n = -3.0$  and  $A = \text{const}$ , where  $A$  is determined mainly by test temperature.

The experiments show that for the P-12 ceramic samples the time to rupture decreases exponentially with increasing temperature (Fig. 2). At the same time for the P-25 samples this dependence is extremal with the peak value occurring at 1450°C: as temperature increases the time to rupture increases initially and then decreases. The apparent activation energy of rupture during creep was determined from the slope of the straight lines (see Fig. 2). For P-12 it was 460 kJ/mole in the experimental temperature range; for P-25 it equals 440 kJ/mole in the range 1450 – 1550°C. We note that the values obtained are close to the creep activation energy [3]. As the crystals increase in size, the time to rupture in the range 1450 – 1550°C increases.

For both types of periclase ceramics, at all testing temperatures the time to rupture decreases as the rate of steady creep increases (Fig. 3). At the same time a linear dependence is observed in logarithmic coordinates with the tangent of the slope angle of the straight lines close to  $-1$ . Thus, the product of the time to rupture by the rate of steady creep remains constant irrespective of the testing temperature, applied load and crystal size. The only exceptions are the results obtained in the experiments performed on comparatively large crystalline samples P-25 at 1400°C. They also fall on a straight line with tangent of the slope angle close to 1 ( $n = -1.3$ ) but appreciably below the ordinate of the general dependence.

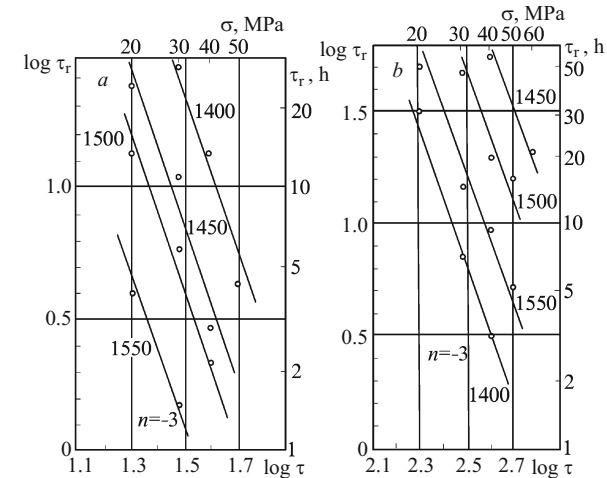


Fig. 1. Time to rupture  $\tau_r$  versus the stress  $\sigma$ : a) P-12; b) P-25 (temperature in °C);  $n$ ) tangent of the slope angle of the straight lines.

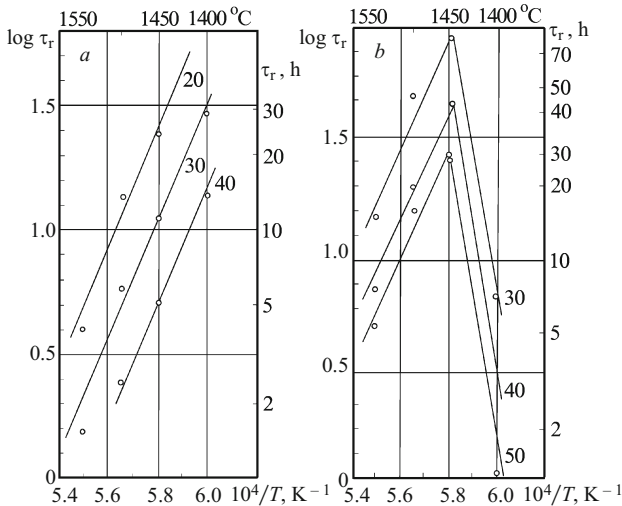
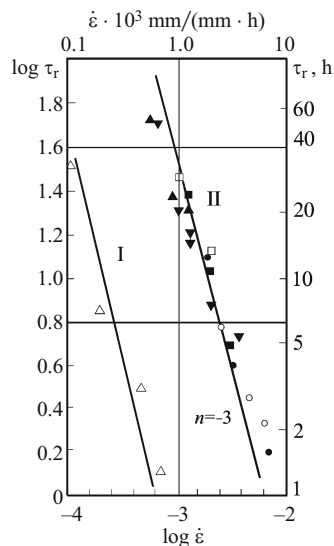


Fig. 2. Time to rupture  $\tau_r$  versus the temperature  $T$ : a) P-12, b) P-25 (the load in MPa is indicated).

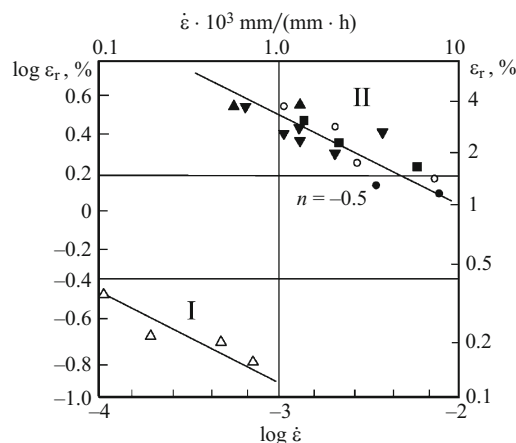
The magnitude of the dip before rupture and the rate of steady creep are related with one another (Fig. 4). A power-law relation with exponent  $n = -0.5$  is observed between them for both types of ceramic. It was also determined that there exists a definite relation between the time and strain before rupture (Fig. 5). It is expressed by a power-law function with the exponent  $n = 4$ . At 1400°C (compared with 1450 – 1550°C) the large crystalline samples exhibit a much smaller dip prior to rupture.

TABLE 1. Characteristics of the Experimental Samples

Batch index	Ceramic type	Predominant crystal size	Apparent density, g/cm <sup>3</sup>	True porosity, %	Bending strength, MPa, at temperature, °C				
					20	1400	1450	1500	1550
P-12	MgO	12	3.47	3.0	120	65	52	45	40
P-25	MgO	25	3.50	2.2	105	60	73	62	55



**Fig. 3.** Relation between the time to rupture  $\tau_r$  and the rate of steady creep  $\dot{\epsilon}$ : I) P-12 at 1400°C; II) general relation.

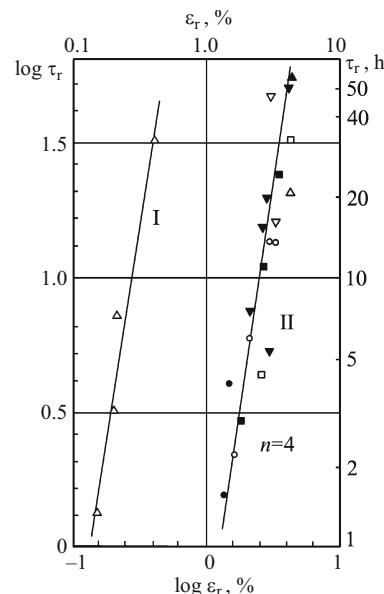


**Fig. 4.** Relation between the dip before rupture  $\epsilon_r$  and the rate of steady creep  $\dot{\epsilon}$ : I) P-12 at 1400°C; II) general relation.

We note one other feature of the plots displayed in Figs. 3 – 5. All experimental points fall practically on a single straight line irrespective of the size of the ceramic crystals and the testing conditions. Only the results obtained for large-crystalline periclase at 1400°C do not follow this behavior; they fall on a separate line with the same slope. It is probable that a different mechanism of strain is observed here; the behavior of the material in this range of testing conditions requires further study.

The magnitude of the dip prior to rupture is also related with the strain. This relation is different for ceramics with different crystal sizes. A significant decrease of strain with increasing load is observed. Thus, in the range 1400 – 1500°C as the stress increases by a factor of 2 so does the strain. However, a definite regularity could not be established.

As temperature increases, the strain prior to rupture increases but no regularity could be established. It should be noted that the maximum strain is observed at 1450°C. An in-



**Fig. 5.** Relation between the time to rupture  $\tau_r$  and the dip prior to rupture  $\epsilon_r$ : I) P-12 at 1400°C; II) general relation.

crease of the stress does not change the dip before rupture significantly.

## CONCLUSIONS

The experimental relations obtained between the characteristics of the creep and creep rupture strength point to the existence of a relation between creep and rupture. The relations found make it possible to determine the dip and time to rupture, while preserving the integrity of the samples, from measurements of the rate of stationary (steady) creep.

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